

4,6-Dichloro-2-((E)-{4-[(E)-3,5-dichloro-2-hydroxybenzylideneamino]butyl-imino}methyl)phenol

Hadi Kargar,^{a*} Reza Kia,^{b‡} Amir Adabi Ardakani^c and Muhammad Nawaz Tahir^d

^aDepartment of Chemistry, Payame Noor University, PO Box 19395-3697 Tehran, I. R. of IRAN, ^bDepartment of Chemistry, Science and Research Branch, Islamic Azad University, Tehran, Iran, ^cArdakan Branch, Islamic Azad University, Ardakan, Iran, and ^dDepartment of Physics, University of Sargodha, Punjab, Pakistan
Correspondence e-mail: h.kargar@pnu.ac.ir, dmntahir_uos@yahoo.com

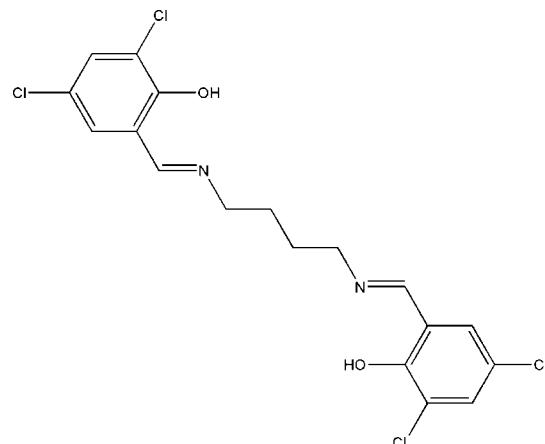
Received 10 June 2012; accepted 25 June 2012

Key indicators: single-crystal X-ray study; $T = 291\text{ K}$; mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$; R factor = 0.045; wR factor = 0.095; data-to-parameter ratio = 17.4.

The asymmetric unit of the title compound, $\text{C}_{18}\text{H}_{16}\text{Cl}_4\text{N}_2\text{O}_2$, comprises half of a potentially tetradeятate Schiff base ligand. It is located about a twofold rotation axis that bisects the central C—C bond of the butane-1,4-diamine group. There are two intramolecular O—H···N hydrogen bonds making *S*(6) ring motifs. In the crystal, molecules are linked by pairs of weak C—H···Cl interactions, forming inversion dimers, which are further connected by C—H···O hydrogen bonds into two-dimensional frameworks that lie parallel to (001).

Related literature

For standard bond lengths, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related Schiff base ligands, see: Kargar *et al.* (2011); Kia *et al.* (2010).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{16}\text{Cl}_4\text{N}_2\text{O}_2$
 $M_r = 434.13$
Orthorhombic, $Pbcn$
 $a = 15.871 (3)\text{ \AA}$
 $b = 12.505 (3)\text{ \AA}$
 $c = 9.4133 (18)\text{ \AA}$

$V = 1868.3 (6)\text{ \AA}^3$

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.65\text{ mm}^{-1}$

$T = 291\text{ K}$

$0.35 \times 0.16 \times 0.14\text{ mm}$

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
 $T_{\min} = 0.805$, $T_{\max} = 0.915$

8772 measured reflections
2057 independent reflections
1264 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.058$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.095$
 $S = 1.02$
2057 reflections

118 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.31\text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.27\text{ e \AA}^{-3}$

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
O1—H1···N1	0.95	1.71	2.565 (3)	149
C5—H5···O1 ⁱ	0.93	2.48	3.370 (3)	160
C3—H3···Cl2 ⁱⁱ	0.93	2.83	3.738 (3)	165

Symmetry codes: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, z$; (ii) $-x + 1, -y + 1, -z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

HK and AAA thank PNU for financial support. MNT thanks GC University of Sargodha, Pakistan, for the research facility.

‡ Present address: Structural Dynamics of (Bio)Chemical Systems, Max Planck Institute for Biophysical Chemistry, Am Fassberg 11, 37077 Göttingen, Germany.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2460).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bruker (2005). *APEX2, SAINT and SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Kargar, H., Kia, R., Pahlavani, E. & Tahir, M. N. (2011). *Acta Cryst. E* **67**, o614.
- Kia, R., Kargar, H., Tahir, M. N. & Kianoosh, F. (2010). *Acta Cryst. E* **66**, o2296.
- Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst. D* **65**, 148–155.

supplementary materials

Acta Cryst. (2012). E68, o2244–o2245 [doi:10.1107/S1600536812028693]

4,6-Dichloro-2-((E)-{4-[(E)-3,5-dichloro-2-hydroxybenzylideneamino]butyl-imino}methyl)phenol

Hadi Kargar, Reza Kia, Amir Adabi Ardakani and Muhammad Nawaz Tahir

Comment

In continuation of our work on the crystal structure analyses of Schiff base ligands (Kargar *et al.*, (2011); Kia *et al.*, (2010), we report herein on the crystal structure of the title compound. The asymmetric unit of the title compound, Fig. 1, comprises half of a potentially tetridentate Schiff base ligand. It is located about a two-fold rotation axis that bisects the central C9-C9a bond of the butane-1,4-diamine group. The bond lengths (Allen *et al.*, 1987) and angles are within the normal ranges. The intramolecular O—H···N hydrogen bonds make *S*(6) ring motifs (Fig. 2 and Table 1; Bernstein *et al.*, 1995).

In the crystal, molecules are linked by pairs of weak C—H···Cl interactions to form inversion dimers which are further connected by C—H···O hydrogen bonds along the *b* axis direction, forming two dimensional networks that lie parallel to the *ab* plane (Table 1 and Fig. 2).

Experimental

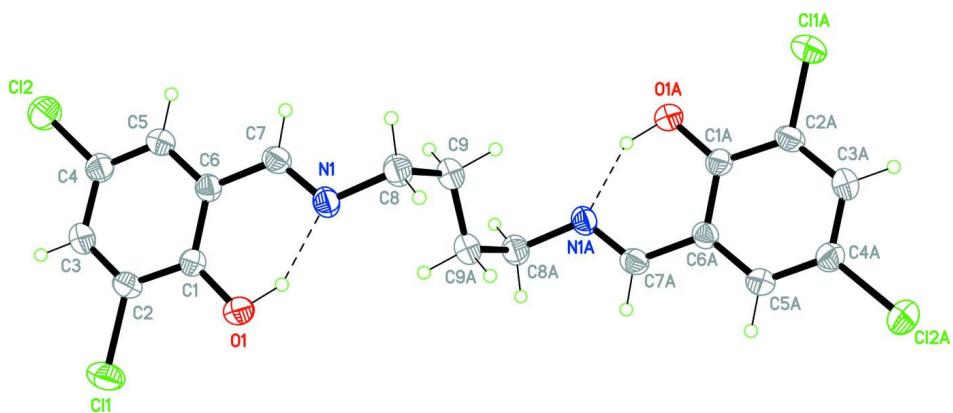
The title compound was synthesized by adding 3,5-dichlorosalicylaldehyde (2 mmol) to a solution of butylenediamine (1 mmol) in ethanol (30 ml). The mixture was refluxed with stirring for 30 min. The resultant solution was filtered. Yellow-needle single crystals of the title compound, suitable for *X*-ray structure determination, were obtained by recrystallization from ethanol by slow evaporation of the solvents at room temperature over several days.

Refinement

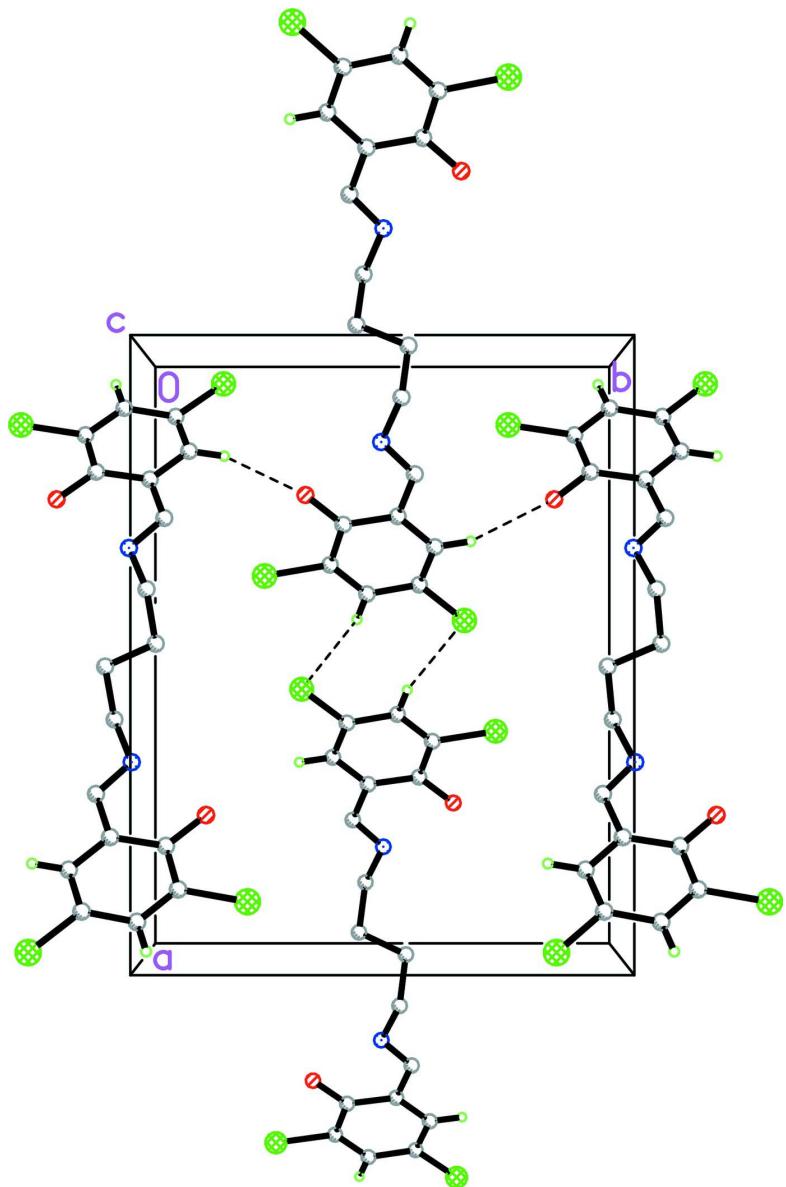
The OH H atom was located in a difference Fourier map and was constrained to ride on the parent O atom with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. The C-bound H-atoms were included in calculated positions and treated as riding atoms: C—H = 0.93 and 0.97 Å for CH and CH₂ H-atoms, respectively, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005)'; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

**Figure 1**

The molecular structure of the title compound, showing 40% probability displacement ellipsoids and the atomic numbering [symmetry code for suffix A = $-x$, $-y + 1$, $-z + 1$].

**Figure 2**

The crystal packing of the title compound showing the C—H···O and weak C—H···Cl interactions (dashed lines; see Table 1 for details) forming two dimensional networks. Only the H atoms involved in hydrogen bonding are shown.

4,6-Dichloro-2-((*E*)-{4-[(*E*)-3,5-dichloro-2-hydroxybenzylideneamino]butylimino}methyl)phenol

Crystal data

$C_{18}H_{16}Cl_4N_2O_2$
 $M_r = 434.13$
Orthorhombic, $Pbcn$
Hall symbol: -P 2n 2ab
 $a = 15.871 (3)$ Å
 $b = 12.505 (3)$ Å
 $c = 9.4133 (18)$ Å
 $V = 1868.3 (6)$ Å³
 $Z = 4$

$F(000) = 888$
 $D_x = 1.543$ Mg m⁻³
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 851 reflections
 $\theta = 2.8\text{--}27.5^\circ$
 $\mu = 0.65$ mm⁻¹
 $T = 291$ K
Needle, yellow
 $0.35 \times 0.16 \times 0.14$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
 $T_{\min} = 0.805$, $T_{\max} = 0.915$

8772 measured reflections
 2057 independent reflections
 1264 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.058$
 $\theta_{\max} = 27.1^\circ$, $\theta_{\min} = 2.1^\circ$
 $h = -20 \rightarrow 12$
 $k = -16 \rightarrow 15$
 $l = -10 \rightarrow 12$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.095$
 $S = 1.02$
 2057 reflections
 118 parameters
 0 restraints
 Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
 Hydrogen site location: inferred from neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0259P)^2 + 0.896P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.31 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F, with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.37204 (6)	0.74164 (6)	0.18745 (9)	0.0673 (3)
Cl2	0.44335 (5)	0.32968 (6)	0.08251 (9)	0.0613 (3)
O1	0.24660 (12)	0.65449 (13)	0.3845 (2)	0.0492 (5)
H1	0.2062	0.6189	0.4413	0.074*
N1	0.16734 (13)	0.50222 (17)	0.5089 (2)	0.0383 (5)
C1	0.29214 (16)	0.57913 (19)	0.3202 (3)	0.0356 (6)
C2	0.35462 (17)	0.60699 (19)	0.2224 (3)	0.0404 (7)
C3	0.40082 (17)	0.5318 (2)	0.1515 (3)	0.0428 (7)
H3	0.4418	0.5526	0.0866	0.051*
C4	0.38606 (17)	0.4244 (2)	0.1772 (3)	0.0413 (7)
C5	0.32680 (17)	0.3932 (2)	0.2738 (3)	0.0402 (7)
H5	0.3181	0.3209	0.2910	0.048*
C6	0.27940 (16)	0.46897 (19)	0.3465 (3)	0.0344 (6)
C7	0.21558 (16)	0.4352 (2)	0.4470 (3)	0.0379 (6)
H7	0.2096	0.3628	0.4670	0.046*
C8	0.10183 (16)	0.4624 (2)	0.6055 (3)	0.0435 (7)
H8A	0.0946	0.5125	0.6833	0.052*

H8B	0.1194	0.3944	0.6452	0.052*
C9	0.01787 (17)	0.4483 (2)	0.5280 (3)	0.0481 (8)
H9A	0.0258	0.3993	0.4492	0.058*
H9B	-0.0225	0.4163	0.5926	0.058*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0782 (6)	0.0361 (4)	0.0874 (6)	-0.0057 (4)	0.0253 (5)	0.0114 (4)
Cl2	0.0628 (5)	0.0484 (4)	0.0727 (6)	0.0047 (4)	0.0223 (4)	-0.0103 (4)
O1	0.0497 (12)	0.0349 (10)	0.0630 (12)	0.0051 (9)	0.0161 (11)	0.0044 (9)
N1	0.0308 (12)	0.0419 (12)	0.0421 (13)	-0.0032 (11)	0.0016 (11)	0.0050 (11)
C1	0.0305 (15)	0.0343 (13)	0.0420 (15)	0.0009 (12)	-0.0014 (13)	0.0019 (12)
C2	0.0410 (17)	0.0321 (13)	0.0481 (17)	-0.0052 (13)	0.0009 (14)	0.0058 (12)
C3	0.0362 (16)	0.0458 (16)	0.0465 (17)	-0.0041 (13)	0.0077 (14)	0.0027 (13)
C4	0.0394 (17)	0.0371 (14)	0.0475 (16)	-0.0003 (13)	0.0042 (14)	-0.0056 (13)
C5	0.0408 (16)	0.0324 (13)	0.0473 (17)	-0.0022 (12)	0.0036 (14)	0.0013 (12)
C6	0.0309 (15)	0.0353 (13)	0.0371 (15)	-0.0014 (12)	-0.0007 (12)	0.0026 (11)
C7	0.0354 (16)	0.0330 (13)	0.0453 (16)	-0.0042 (13)	-0.0035 (13)	0.0041 (12)
C8	0.0383 (16)	0.0475 (16)	0.0446 (16)	-0.0011 (14)	0.0032 (15)	0.0056 (13)
C9	0.0408 (18)	0.0456 (16)	0.0578 (19)	-0.0038 (14)	0.0128 (16)	0.0048 (14)

Geometric parameters (\AA , ^\circ)

Cl1—C2	1.738 (2)	C4—C5	1.365 (3)
Cl2—C4	1.739 (3)	C5—C6	1.390 (3)
O1—C1	1.333 (3)	C5—H5	0.9300
O1—H1	0.9457	C6—C7	1.449 (3)
N1—C7	1.276 (3)	C7—H7	0.9300
N1—C8	1.468 (3)	C8—C9	1.530 (4)
C1—C2	1.397 (4)	C8—H8A	0.9700
C1—C6	1.414 (3)	C8—H8B	0.9700
C2—C3	1.366 (4)	C9—C9 ⁱ	1.506 (5)
C3—C4	1.385 (3)	C9—H9A	0.9700
C3—H3	0.9300	C9—H9B	0.9700
C1—O1—H1	106.9	C5—C6—C7	120.1 (2)
C7—N1—C8	119.0 (2)	C1—C6—C7	119.9 (2)
O1—C1—C2	120.5 (2)	N1—C7—C6	121.8 (2)
O1—C1—C6	122.1 (2)	N1—C7—H7	119.1
C2—C1—C6	117.4 (2)	C6—C7—H7	119.1
C3—C2—C1	122.1 (2)	N1—C8—C9	111.1 (2)
C3—C2—Cl1	119.3 (2)	N1—C8—H8A	109.4
C1—C2—Cl1	118.6 (2)	C9—C8—H8A	109.4
C2—C3—C4	119.4 (3)	N1—C8—H8B	109.4
C2—C3—H3	120.3	C9—C8—H8B	109.4
C4—C3—H3	120.3	H8A—C8—H8B	108.0
C5—C4—C3	120.7 (2)	C9 ⁱ —C9—C8	113.3 (3)
C5—C4—Cl2	120.5 (2)	C9 ⁱ —C9—H9A	108.9
C3—C4—Cl2	118.9 (2)	C8—C9—H9A	108.9

C4—C5—C6	120.4 (2)	C9 ⁱ —C9—H9B	108.9
C4—C5—H5	119.8	C8—C9—H9B	108.9
C6—C5—H5	119.8	H9A—C9—H9B	107.7
C5—C6—C1	120.0 (2)		
O1—C1—C2—C3	-178.1 (2)	C4—C5—C6—C7	178.9 (2)
C6—C1—C2—C3	1.5 (4)	O1—C1—C6—C5	178.1 (2)
O1—C1—C2—Cl1	0.4 (4)	C2—C1—C6—C5	-1.5 (4)
C6—C1—C2—Cl1	-180.0 (2)	O1—C1—C6—C7	-0.5 (4)
C1—C2—C3—C4	-0.4 (4)	C2—C1—C6—C7	179.9 (2)
Cl1—C2—C3—C4	-178.8 (2)	C8—N1—C7—C6	177.7 (2)
C2—C3—C4—C5	-0.9 (4)	C5—C6—C7—N1	-175.5 (2)
C2—C3—C4—Cl2	178.2 (2)	C1—C6—C7—N1	3.1 (4)
C3—C4—C5—C6	1.0 (4)	C7—N1—C8—C9	-93.8 (3)
Cl2—C4—C5—C6	-178.2 (2)	N1—C8—C9—C9 ⁱ	-63.4 (4)
C4—C5—C6—C1	0.3 (4)		

Symmetry code: (i) $-x, -y+1, -z+1$.

Hydrogen-bond geometry (\AA , $^{\circ}$)

$D\text{—H}\cdots A$	$D\text{—H}$	$\text{H}\cdots A$	$D\cdots A$	$D\text{—H}\cdots A$
O1—H1 \cdots N1	0.95	1.71	2.565 (3)	149
C5—H5 \cdots O1 ⁱⁱ	0.93	2.48	3.370 (3)	160
C3—H3 \cdots Cl2 ⁱⁱⁱ	0.93	2.83	3.738 (3)	165

Symmetry codes: (ii) $-x+1/2, y-1/2, z$; (iii) $-x+1, -y+1, -z$.